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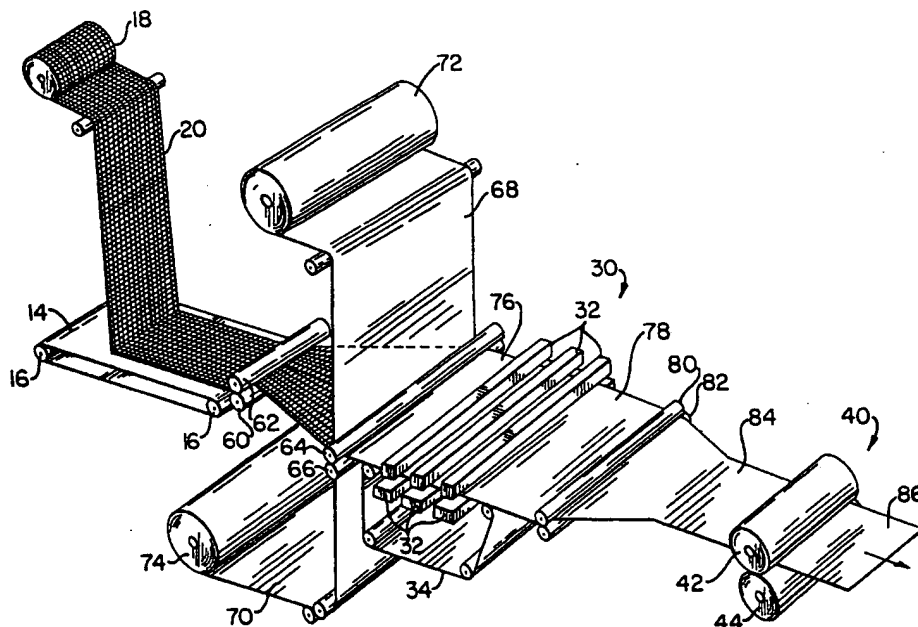
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(54) Title: COMPOSITE ELASTIC NONWOVEN FABRIC



(57) Abstract

The invention provides composite elastic nonwoven fabrics and the process of making them. The composite elastic fabrics of the invention include an elastomeric net and at least one fibrous web including binder fibers and which is intimately hydroentangled with the elastomeric net. At least a portion of the binder fibers in the hydroentangled fibrous web have been thermally activated thereby bonding the hydroentangled web into a coherent, substantially unitary structure encompassing the elastomeric net.

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## COMPOSITE ELASTIC NONWOVEN FABRIC

### Field of the Invention

The invention relates to composite elastic nonwoven fabrics and to processes for producing them. More specifically, the invention relates composite  
5 nonwoven fabrics having desirable coherency, strength, conformability, and stretch and recovery properties.

### Background of the Invention

Nonwoven elastic fabrics have been the subject of considerable attention and effort. Elastic  
10 fabrics are desirable for use in bandaging materials, garments, diapers, supportive clothing and personal hygiene products because of their ability to conform to irregular shapes and to allow more freedom of body movement than fabrics with limited extensibility.

15 Elastomeric materials have been incorporated into various fabric structures to provide stretchable fabrics. In many instances, such as where the fabrics are made by knitting or weaving, there is a relatively high cost associated with the fabric. In cases where  
20 the fabrics are made using nonwoven technologies, the fabrics can suffer from insufficient strength and only limited stretch and recovery properties.

Elastomers used to fabricate elastic fabrics often have an undesirable rubbery feel. This is  
25 particularly true with thermoplastic elastomers rather than crosslinked elastomers. When these materials are used in composite nonwoven fabrics, the hand and

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texture of the fabric can be perceived by the user as sticky or rubbery and therefore undesirable.

U.S. Patent 4,775,579 to Hagy, et al.

discloses desirable composite elastic nonwoven fabrics  
5 containing staple textile fibers intimately  
hydroentangled with an elastic web or an elastic net.  
One or more webs of staple textile fibers and/or wood  
pulp fibers can be hydroentangled with an elastic net  
according to the disclosure of this invention. The  
10 resulting composite fabric exhibits characteristics  
comparable to those of knit textile cloth and possesses  
superior softness and extensibility properties. The  
rubbery feel traditionally associated with elastomeric  
materials can be minimized or eliminated in these  
15 fabrics.

U.S. 4,413,623 to Pieniak discloses a  
laminated structure such as a disposable diaper which  
can incorporate an elastic net into portions of the  
structure. The elastic net can be inserted in a  
20 stretched condition between first and second layers of  
the structure and bonded to the layers while in the  
stretched condition. Subsequent relaxation of the  
elastic net can result in gathering of the structure.

U.S. 4,525,407 to Ness discloses elastic  
25 fabrics which include an elastic member, which may be  
an elastic net, intermittently bonded to a substrate  
which prior to stretching is less easily extensible  
than the elastic member. The nonelastic member is  
bonded to the elastic member and the entire composite  
30 is rendered elastic by stretching and relaxation.

U.S. 4,606,964 to Wideman discloses a bulked  
composite web which can be prepared by bonding a  
gatherable web to a differentially stretched elastic  
net. Subsequent relaxation of the differentially  
35 stretched net is said to result in gathering of the  
fabric.

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These and other elastic nonwoven fabrics and the processes for manufacturing them can suffer from various disadvantages. In some instances, the fibers are not firmly anchored into the composite web so that following repeated stretch and relaxation, fiber shedding and pilling can be problematic. In other instances, the nonwoven net is insufficiently integrated into the composite fibrous structure so that separation of the net can occur and/or the fabric can lose elastic properties. In other instances, the fabric exhibits a low extensibility which is well below the possible extensibility afforded by the elastic net. In addition, the manufacturing processes associated with prior art fabrics can involve complicated and difficult manufacturing steps increasing the cost of the fabric and/or decreasing the fabric uniformity.

#### Summary of the Invention

The invention provides composite elastic nonwoven fabrics which are coherent and exhibit improved strength and elasticity properties. The fabrics can have a high degree of elasticity and stretch recovery while fiber shedding, pilling or fuzzing problems are minimized or eliminated. The fabrics can be provided with desirable softness characteristics. In various embodiments of the invention, differential elasticity can be provided in the fabrics of the invention.

The composite elastic nonwoven fabrics of the invention include an elastomeric net and a fibrous web intimately hydroentangled together. The fibrous web includes binder fibers and at least a portion of the binder fibers extend through apertures in the elastomeric net. The binder fibers in the hydroentangled fibrous web have been thermally activated to thereby bond the hydroentangled fibrous web into a coherent, substantially unitary structure encompassing the elastomeric net. Typically the binder

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fibers bond to themselves and may or may not bond to other fibers or to the net in the structure.

The composite nonwoven elastic fabrics of the invention can be manufactured by relatively simple and straightforward manufacturing processes which involve forming a layered structure including the binder fiber-containing fibrous web and the elastomeric net; hydroentangling the layered structure; and then thermally treating the hydroentangled fabric.

10 Hydroentangling and bonding can be accomplished with or without stretching of the elastic net to provide a highly elastic and coherent composite fabric. Thermal bonding can be accomplished employing any of various well known process steps including ultrasonics,

15 calender rolls or through-air bonding, and is preferably accomplished using heated calender rolls or through-air bonding. Although patterned calender rolls can be employed, advantageously smooth calender rolls are employed for bonding the composite web.

20 Various textile staple fibers such as polyolefins, polyesters, nylon, and the like, and/or wood pulp fibers are preferably included in the fibrous web which is hydroentangled with the elastomeric net. These fibers provide desirable aesthetic qualities to the composite fabric. The elastomeric net is advantageously maintained within the interior of the fabric so that the fabric does not have a rubbery feel or hand. Because binder fibers are incorporated into the fibrous web, the composite fabric possesses a

25 unitary coherent structure and the textile and/or wood pulp fibers are firmly anchored in the composite web.

In preferred embodiments of the invention, separate fibrous webs containing binder fibers are disposed on opposite sides of the elastomeric net prior to hydroentangling. This ensures that the elastomeric net is confined within the interior of the composite fabric and that sufficient textile fibers are provided

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on each side of the elastomeric net so that the hand and coherent nature of the fabric is improved.

Additionally, it is preferred that less than about 50 wt. % of the fibers of the fibrous layer or  
5 layers are binder fibers so that the composite elastic fabric resulting from thermal treating has improved drapability and softness. Various binder fibers can be employed in the invention. Preferably, bicomponent  
10 fibers are employed as binder fibers in the fabrics of the invention.

The invention also provides fabrics having anisotropic stretch properties, i.e., directionalized stretch properties, by employing elastomeric nets  
15 having different stretch properties in the longitudinal net strands and the transverse net strands. Thus, elastomeric nets in which only the strands of one direction, e.g., only the longitudinal, or only the transverse strands, are elastomeric, can be employed to  
20 provide composite fabrics having elasticity in only the longitudinal or only the transverse dimension, respectively. Alternatively, elastomeric materials having different mechanical properties can be employed for the longitudinal and transverse elastomeric net  
25 strands.

#### Brief Description of the Drawings

In the drawings which form a portion of the original disclosure of the invention:

Figure 1 schematically illustrates one  
30 preferred method and apparatus for manufacturing a preferred composite elastic nonwoven web according to the invention;

Figure 2 schematically illustrates another preferred method and apparatus for the manufacture of  
35 another preferred fabric according to the invention;

Figure 3 illustrates a fragmentary exploded view of preferred intermediate layered structure

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employed in the production of elastic nonwoven fabrics according to the invention; and

Figure 4 illustrates a fragmentary perspective view of a composite fabric of the invention showing the exterior fibrous surface of the fabric and the interior elastomeric net which has been integrated with the fibrous webs shown.

#### Detailed Description of the Invention

In the following detailed description of the invention, specific preferred embodiments of the invention are described to enable a full and complete understanding of the invention. It will be recognized that it is not intended to limit the invention to the particular preferred embodiments described, and although specific terms are employed in describing the invention, such terms are used in the descriptive sense for the purpose of illustration and not for the purpose of limitation. It will be apparent that the invention is susceptible to variation and changes within the spirit of the teachings herein.

Figure 1 schematically illustrates a preferred process and apparatus for forming the composite nonwoven webs of the invention. A carding apparatus 10 forms a first carded layer 12 onto forming screen 14. Carded fibrous layer 12 includes binder fibers and preferably also includes synthetic or natural fibers. As explained in greater detail later the binder fibers are advantageously present in an amount of between about 10 and 50 wt. % in carded layer 12. Web 12 is moved by forming screen 14 in the longitudinal direction by rolls 16.

A conventional supply system including a supply roll 18 deposits an elastomeric net 20 onto carded layer 12. The elastomeric net includes spaced apart longitudinal and transverse strands which intersect to form apertures. In addition, irregular geometry nets such as diamond nets and the like can be



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used in place of net 20. With a rectangular net, for example, one or both of the longitudinal or transverse groups of strands are formed of a thermoplastic or crosslinked elastomeric material such as a polyurethane, a styrene triblock copolymer, or the like, as discussed in greater detail later. Preferably, the longitudinal and transverse strands are provided in an amount such that there are between about 5 and about 30 or more strands per inch.

10 As the net 20 is deposited onto carded web 12, a two layer structure 22 is formed and is conveyed by forming screen 14 in the longitudinal direction as indicated in Figure 1. A second carding apparatus 24 deposits a second carded fibrous layer preferably  
15 comprising binder fibers onto the composite layered structure 22 to thereby form a three-layer composite structure 28 consisting of a carded web/elastomeric net/carded web. The binder fibers and other fibers making up carded web 26 can be the same or different as  
20 compared to the fibers in carded web 12. The content of binder fibers in carded web 26 can be the same or different as compared to the content of binder fibers in carded web 12.

The three-layer composite web 28 is conveyed  
25 longitudinally as shown in Figure 1 to a hydroentangling station 30 wherein a plurality of manifolds 32, each including one or more rows of fine orifices, direct high pressure jets through the composite web 28 to intimately hydroentangle the fibers  
30 in the webs 12 and 26 with each other and with elastomeric net 20. As a result of the hydroentangling treatment, at least a portion of the fibers in each of the carded layers 12 and 26 extend through apertures in the elastomeric net and into the carded layer on the  
35 other side of the net.

The hydroentangling station 30 is constructed in a conventional manner as known to the skilled

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artisan and as described, for example, in U.S. Patent 3,485,706 to Evans, which is hereby incorporated by reference. As known to the skilled artisan, fiber hydroentanglement is accomplished by jetting liquid, typically water, supplied at a pressure from about 200 psig up to about 1800 psig or greater, to form fine, essentially columnar liquid streams. The high pressure liquid streams are directed to at least one surface of the composite layered structure. The composite is supported on a foraminous support stream 34 which can have a pattern to form a nonwoven structure with a pattern or with apertures, or the screen can be designed and arranged to form a hydraulically entangled composite which is not patterned or apertured. The laminate can be passed through a second hydraulic entangling station to enable hydraulic entanglement on the other side of the composite web fabric.

During the hydroentanglement treatment, the binder fibers and the staple or natural fibers, when present, in carded web layers 12 and 26 are forced into and/or through the elastomeric net 20. Preferably, the hydroentangling treatment is sufficient to force at least a portion of the binder fibers present in at least one of the layers 12 and 26 into and/or through the apertures in the elastomeric net 20. More preferably, the hydroentangling treatment is sufficient to force at least a portion of the binder fibers in both carded layers 12 and 26 into and/or through the apertures in the elastomeric net 20. It is also desirable and preferred that other fibers, when present, such as staple and/or natural fibers in webs 12 and 26 are forced into and through the apertures of the elastomeric web 20.

The elastomeric web remains in a substantially planer arrangement during the hydroentangling treatment. Thus, the longitudinal and transverse strands, i.e. machine direction (MD) and

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cross machine direction (CD) strands, respectively, of the elastomeric net 20 undergo little if any movement in the cross-sectional direction, i.e. the Z-direction, within the web. Thus, the elastomeric net remains in a discrete interior cross-sectional portion of the composite web.

A condensed, hydraulically entangled composite web 36 exits the hydroentanglement station 30, and is dried at a conventional drying station (not shown) and is then directed into a thermal treatment station 40, shown in Figure 1 as heated calender rolls 42 and 44. The operating temperature of heated rolls 42 and 44 should be adjusted to a surface temperature such that the binder fibers present in the composite web 36 are thermally activated to bind the composite web into a coherent, unitary structure. On the other hand, the heat transfer conditions are advantageously maintained to avoid thermal degradation or melting of the elastomeric net 20 which is present within the interior of the composite web 36. The bonding conditions can, in some instances depending on the fibers and net used, be controlled to obtain fiber bonding to the net, while simultaneously avoiding thermal degradation of the elastomer or its stretch and recovery properties.

While softening of the elastomeric net 20 can, in some instances, be desirable and helpful for bonding of the binder fibers and/or staple fibers in the composite web to the net, melting of the elastomeric net is advantageously avoided. While the bonding between the binder fibers and/or other natural or staple fibers present in the composite web 36 to the elastomeric net 20 can substantially improve the overall coherency and unitary structure of the ultimate thermally bonded web, it has been found that melting and/or thermally degrading the elastomeric net 20

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decreases both the strength and elasticity of the thermally bonded web.

A thermally-bonded composite elastic web 46 is removed from the nip of the heated rolls 42 and 44 and wound by conventional means onto roll 50. The composite elastic web 48 can be stored on roll 50 or immediately passed to end use manufacturing processes, for example for use in bandages, diapers, disposable undergarments, personal hygiene products and the like.

The method illustrated in Figure 1 is susceptible to numerous preferred variations. For example, although the schematic illustration of Figure 1 shows carded webs being formed directly during the in-line process, it will be apparent that the carded webs can be preformed and supplied as rolls of preformed webs. Similarly, although the elastomeric net is shown being supplied as a roll of a preformed net, the net can be formed directly in-line. Similarly, although Figure 1 illustrates use of fibrous webs 12 and 26 both above and below the elastomeric net 20, only a single fibrous web such as web 12 can be employed or more than two fibrous webs can be employed.

The heated calender rolls 42 and 44 can, in other embodiments of the invention, be replaced by other thermal activation zones, for example in the form of a through-air bonding oven or in the form of a microwave or other RF treatment zone. An especially preferred through-air bonding treatment employs support screens both above and below the fabric and contacting both surfaces thereof during passage through the oven. The screens are advantageously metallic screens resulting in conductive heating of both fabric surfaces by contact with the upper and lower metal screens, respectively. Other heating stations such as ultrasonic welding stations can also be advantageously used in the invention. Such conventional heating stations are known to those skilled in the art and are

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capable of effecting substantial thermal fusion of the binder fibers present in the composite web 36.

Nonwoven webs other than carded webs are also advantageously employed in the production of fabrics of the invention. Nonwoven staple webs can be formed by air laying, garnetting, wet laying and similar processes known in the art. Spunbonded webs which are extensible in the MD or CD direction because of little or no filament-to-filament bonding can be substituted for either or both of carded webs 12 and 26 and/or can be used in combination with one or both of carded webs 12 and 26. Similarly, meltblown webs which are extensible in either or both of the MD and CD direction can be substituted for either of carded webs 12 and 26 and/or can be used in conjunction with one or both of carded webs 12 and 26.

Figure 2 illustrates a process of the invention wherein elastomeric net 20 is stretched in the CD and/or MD direction during hydroentanglement of the composite fabric. Elastomeric web 20 is deposited onto a screen 14 and fed via a pair of feed rolls 60, 62 to a pair of stretching rolls 64, 66. Rolls 64 and 66 may be operated at a speed greater than rolls 60 and 62 thereby stretching the net 20 in the MD at the same time the net is expanded in the CD. Clamps or a tenter frame are typically required (not shown) for CD stretching to minimize necking.

Two preformed webs 68 and 70 are fed via supply rolls 72 and 74, respectively, to the feed rolls 64 and 66 for layering with the net 20 while it is in the stretched condition. One or both of webs 68 and 70 includes binder fibers, preferably in an amount of 10-50 wt. %. It is also preferred that at least one of webs 68 and 70 is a staple fiber web which can be preformed via air laying, garnetting or carding. In addition, at least one of the webs 68 and 70 can preferably constitute a spunbond or a meltblown web.

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The combined 3-layer structure 76 is passed through hydroentangling station 30 while the net 20 is maintained in a stretched condition by down-stream rollers 80 and 82. Clamps or tenting again may be required if the web is stretched in the CD and tends to neck. High pressure water jets from manifolds 32 force fibers from the fibrous webs 68 and 70 through the apertures of the stretched elastic net 20 during passage through the hydroentangling station.

The hydroentangled and consolidated structure 78 issuing from the hydroentangling station 30 is thereafter allowed to relax and is then dried by conventional means such as an oven (not shown). The thus dried and relaxed composite web 84 is passed through a thermal bonding station 40 comprising heated calender rolls 42 and 44 for thermal activation of the thermal binder fibers in the consolidated web 84. As shown in Figure 2, the thermal treatment of the consolidated web 84 is advantageously conducted while the elastomeric net 20 is in a relaxed condition, although thermal treatment can be conducted while the web is maintained in a stretched condition if desired. As in the previously described process illustrated in Figure 1, the thermal treatment at thermal treating station 40 is advantageously conducted such that heat transfer is sufficient to melt the binder fibers but insufficient to thermally deform the elastomeric net 20. The degree of temperature control which is required will be dependent, in part, on the type of elastomer used in the net. For example, if cross-linked rather than thermoplastic elastomers (TPE's) are used, the need to tightly control heat transfer conditions is less urgent.

The thermally treated coherent web 86 issuing from the heated calender rolls 42 and 44 is passed to storage or end use applications.

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The process illustrated in Figure 2 is particularly advantageous for preparing an elastomeric composite web wherein one or more of the webs employed to make the composite are a spunbonded or meltblown web. Because meltblown and spunbonded webs typically have only limited extensibility in either the MD or CD direction, integration of such webs with the elastomeric net while the net is in a stretched condition allows the resultant composite web to have an extensibility following relaxation and thermal treatment, while retaining some of the characteristics of the component fabrics. When spunbonded webs are incorporated, an abrupt increase in the modulus of the composite fabric can be achieved. When the composite is extended to the same degree used in its manufacture, the tensile properties of the spunbond dominate the elastic properties of the net. When meltblown webs are incorporated, the resulting composite retains the high cover and barrier properties associated with meltblown fibers.

It is preferred that when spunbond and/or meltblown webs are employed in the process illustrated in Figure 2 that at least one web combined into the composite web 76 include binder fibers in staple form. The use of binder fibers in staple form allows the binder fibers to become fully integrated through the structure of the elastomeric net and preferably also through the structure of the meltblown or spunbonded web when used. It is also preferred that non-binder staple fibers be also included in one of the nets combined in the hydroentangling station 30 so that the non-binder fibers also are employed for integration of the composite elastomeric web.

As with the process illustrated in Figure 1, the process illustrated in Figure 2 is susceptible to numerous variations. Thus, the thermal treating station 40 can comprise any of the previously described

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thermal treating stations. Likewise, the fibrous webs 68 and 70 can be formed in-line where desirable.

Moreover, although net 20 is shown in Figure 2 as stretched in both the MD and CD directions, stretching  
5 can be carried out in only the MD, or in only the CD direction during consolidation of the net with the fibrous webs 70 and 72 by hydroentanglement.

Additionally although two fibrous webs 68 and 70 are shown in Figure 2, only one, or more than two fibrous  
10 webs can be combined with the stretched net 20 during the hydroentanglement and thermal bonding steps accomplished at stations 30 and 40.

Figure 3 illustrates an exploded view of the three layered structure 28 of Figure 1 prior to  
15 hydroentanglement. At least one of the carded web layers 12 and 26 comprises binder fibers in an amount greater than about 5%, preferably greater than about 10% by weight. Binder fibers are known in the art and include fibers made from low melting polyolefins such  
20 as polyethylenes; polyamides and particularly copolyamides; polyesters and particularly copolyesters; acrylics and the like. The binder fiber may have a higher or lower activation temperature than the melting or softening point of the net. In the case that the  
25 binder fiber activates above the glass transition temperature of the hard segment of the thermoplastic elastomer, then heating conditions must be closely controlled to bind the fibers without deforming or degrading the net.

30 Particularly preferred binder fibers include bicomponent and multi-component fibers such as sheath/core, side-by-side, sectorized or similar bicomponent fibers wherein at least one component of the fiber is a low melting material such as a  
35 polyethylene, a copolyester, a copolyamide, or the like. Particularly preferred bicomponent fibers have a melting temperature for the binder portion of the fiber



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in the range of between about 100 and 135°C. Such fibers include polypropylene/polyethylene and polyester/polyethylene sheath/core fibers and polyester/copolyester sheath/core fibers. One particularly preferred binder fiber is a copolyester/polyester sheath/core fiber having a melting point of about 110°C commercially available from Hoechst-Celanese Corporation as "K-54".

Preferably, each of webs 12 and 26 also include staple and/or natural fibers such as fibers formed from polyester, polyolefins such as polypropylene or polyethylene, nylon, acrylic, modacrylic, rayon, cellulose acetate, biodegradable synthetics such as a biodegradable polyester, aramide, fluorocarbon, polyphenylene sulfide staple fibers and the like. Preferred natural fibers include wool, cotton, wood pulp fibers and the like. Blends of such fibers can also be used. In addition, all or a portion of the staple fibers can be glass, carbon fibers or the like.

As indicated previously, the binder fibers are preferably present in an amount of between about 10 and 50 wt. %, more preferably between about 20 and about 40 wt. %, most preferably between about 25 and 35 wt. %. The content of the binder fiber is adjusted to provide coherency to the overall combined web without adding an undesirably stiff or boardy feeling to the web. The specific content of the binder fiber will be dependent, at least to some extent, on the type of binder fiber used and on the type of staple fiber used.

The elastic net 20 includes an elastic material making up either or both of the longitudinal, i.e. machine direction, strands and/or the transverse, i.e. cross machine direction, strands, (assuming the net is rectangular). Suitable elastomers include the diblock and triblock copolymers based on polystyrene (S) and unsaturated or fully hydrogenated rubber

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blocks. The rubber blocks can consist of butadiene (B), isoprene (I), or the hydrogenated version, ethylene-butylene (EB). Thus, S-B, S-I, S-EB, as well as S-B-S, S-I-S, and S-EB-S block copolymers can be used. Preferred elastomers of this type include the KRATON polymers sold by Shell Chemical Company or the VECTOR polymers sold by DEXCO. Other elastomeric thermoplastic polymers include polyurethane elastomeric materials such as ESTANE sold by BF Goodrich Company; polyester elastomers such as HYTREL sold by E.I. Du Pont De Nemours Company; polyetherester elastomeric materials such as ARNITEL sold by Akzo Plastics; and polyetheramide elastomeric materials such as PEBAX sold by ATO Cheme Company.

The elastic strands in the elastomeric net can also be prepared from blends of thermoplastic elastomers with other polymers such as polyolefin polymers, e.g. blends of KRATON polymers with polyolefins such as polypropylene and polyethylene, and the like. These polymers can provide lubrication and decrease melt viscosity, allow for lower melt pressures and temperatures and/or increase throughput, and provide better bonding properties too. In a particularly preferred embodiment of the invention, polymers can be included in the blend as a minor component, for example in an amount of from about 5% by weight up to about 50% by weight, preferably from about 10 to about 30% by weight. Suitable thermoplastic materials include poly(ethylene-vinyl acetate) polymers having an ethylene content of up to about 50% by weight, preferably between about 15 and about 30% by weight, and copolymers of ethylene and acrylic acid or esters thereof, such as poly(ethylene-methyl acrylate) or poly(ethylene-ethyl acrylate) wherein the acrylic acid or ester component ranges from about 5 to about 50% by weight, preferably from about 15 to 30% by weight.

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As indicated previously, the degree of elasticity of the longitudinal and transverse strands of a rectangular elastomeric net can be the same or different. Fabrics having differential stretch in the longitudinal and transverse directions can be provided in accordance with the invention by employing strands in the elastomeric net in either the transverse or longitudinal direction which have little or no elasticity.

In many instances, it is desirable that the number of strands per inch in the longitudinal and transverse dimensions be different. Generally, there are between about 2 to about 30 strands per inch in both the longitudinal and transverse directions although greater numbers of strands can be employed where desirable. In some instances, the fabric of the invention can be used in articles wherein an edge of the fabric is exposed. In such instances it can be desirable to minimize the diameter of the strands which will be exposed along the cut edge of the fabric. Generally, the elastomeric nets used in the invention will have a basis weight ranging from about 15 grams per square meter, to about 200 grams per square meter, more preferably from about 35 to about 75 grams per square meter and can employ strands having diameters ranging from 50 to 600 microns.

As indicated previously, the fabrics of the invention can also incorporate spunbonded nonwovens, including polyolefin, nylon, polyester, copolymers of the same and other such webs as are known to those skilled in the art. Similarly, meltblown nonwovens including both elastomeric and non-elastomeric meltblown webs prepared from polyolefins, nylon, polyesters, random and block copolymers, elastomers and the like are also employed in fabrics of the invention.

Figure 4 illustrates an exploded fragmentary view of a fabric according to the invention. As

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illustrated in Figure 4, the elastomeric net is fully encompassed within the fibrous portion of the composite web. The fibers of the fibrous portion of the web extend through the apertures in the net and thus the fabric is a unitary coherent fabric. Because the binder fibers bind the fibers in the fibrous web together, the fabric is not prone to delamination and problems with fuzziness and lintiness can be avoided. Despite the use of binder fibers the composite fabric possesses substantial elasticity. The elastomeric net does not contribute a rubbery like feel to the surface of the fabric because the net is maintained within the interior of the fabric.

The following examples are provided to illustrate the fabrics of the invention and processes for making them but are not to be construed as limitations on the invention.

In all examples set forth below a styrene-butadiene-styrene triblock thermoplastic elastomer, sold under the trade name KRATON D 2104 by Shell Chemical, was used as the elastomer in the elastic net.

#### EXAMPLES 1 THROUGH 5

These examples demonstrate the effects of bonding and pre-stretching on fabric properties. All the samples were produced with a fiber blend of:

- 70 wt. % 2.2 dpf x 1.5" Type-182 Herculon® polypropylene staple fiber from Hercules,
- 30 wt. % 2.0 dpf x 1.5" K-54 copolyester/polyester bicomponent fiber from Hoechst Celanese Corp.

The composites all consist of a sandwich with: a 33 g/m<sup>2</sup> basis weight layer of carded, blended fiber; a 170 g/m<sup>2</sup> basis weight 18 x 9 (strands/inch, MD x CD) rectangular elastic net; and an additional 33 g/m<sup>2</sup> layer of carded, blended fiber. The total fiber basis weight in the composite is 66 g/m<sup>2</sup>.

All samples were entangled on a 31 x 25 (strands/inch) screen at 240 ft/min with two passes at

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400 psi and four passes at 1400 psi for each side. The examples differ by thermal treatment and pre-stretching.

Examples 1, 2 and 3 were not pre-stretched; 5 they were hydroentangled with the net under minimal tension and in its relaxed state. The samples were then dried prior to the thermal treatments set forth below.

In Examples 4 and 5 the net was pre-stretched 10 100% in the MD and was entangled in the stretched state. After entanglement, the composite fabric was relaxed and dried. It was then ready for thermal treatment as set forth below.

The fabrics of Examples 1 and 4 were not 15 thermally treated while the fabrics of Examples 2, 3 and 5 were. The fabrics of Examples 2 and 5 were thermally treated through a double-wire through-air bonding oven at 155°C. The fabric of Example 3 was 20 thermally treated by calendering with two smooth rolls at 100 ft/min, with a calender gap of 0.023", and calender roll temperature of 135°C for each roll.

The properties of these fabrics are shown in Table 1, below. Example 1 was un-bonded and could be stretched readily in both directions. It was easy to 25 delaminate however, with a peel strength of only 316 grams. Example 2, on the other hand, was through-air bonded. It was very difficult to delaminate. The peel strength was over 5000 g, and there was adhesive failure in the peel tape prior to a cohesive failure of 30 the fabric. This result clearly demonstrates how bonding gives a coherent structure.

Example 3 was unstretched and calender-bonded. It too had a higher peel strength than Example 1, but not nearly as high as Example 2 (through-air 35 bonded). Examples 2 and 3 both stretched easily in the cross-machine direction (CD), but due to fiber orientation and bonding, were much more resistant to

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stretching in the machine direction (MD), as demonstrated in MD tensile strength results.

Finally, Examples 4 and 5 were pre-stretched 100% in the MD prior to hydroentanglement. Both the bonded and un-bonded versions were easy to stretch in both directions. Another benefit of bonding is demonstrated here; namely, fabric toughness. The bonded version was much tougher, and required more force to elongate than the un-bonded version.

10 All bonded fabrics have less propensity to pill, fuzz, and/or shed fibers.

Table 1 provides a summary of the fabrics and their associated mechanical properties.

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Table 1. Description and Properties of Examples 1 Through 5.

ITEM		EXAMPLE									
		1		2		3		4		5	
Pre-stretch		none		none		none		100% MD		100% MD	
Heat Treatment		none		through-air		calender		none		through-air	
Strip Tensile Test:		MD	CD	MD	CD	MD	CD	MD	CD	MD	CD
Load (g/in) @ 25% el.		472	172	2197	272	656	190	121	174	212	299
Load (g/in) @ 50% el.		1174	239	2991	459	1752	264	185	252	357	615
Load (g/in) @ 75% el.		1487	295	2569	585	2252	313	253	317	700	904
Load (g/in) @ 100% el.		1029	325	1678	680	2078	355	356	386	1206	1115
Peak Load (g/in)		2157	2251	3103	2183	2292	1922	2757	2163	3006	2308
Elongation (%) at Peak		963	1005	61	1002	83	1033	317	781	237	897
Cycle Test: Load (g/in) at 50% Elongation	Pull 1	930	261	2898	482	2109	273	201	258	407	676
	Pull 2	530	294	1910	403	1168	245	188	228	322	554
	Pull 3	433	223	1614	380	858	238	182	222	320	514
% Set @ 50% el.		3.5%	3.3%	4.3%	3.6%	3.8%	3.4%	3.6%	3.5%	3.7%	3.8%
Cycle Test: Load (g/in) at 100% Elongation	Pull 1	1749	335	2814	681	1160	372	406	404	1466	989
	Pull 2	585	294	874	551	598	325	348	343	1050	755
	Pull 3	484	285	671	502	454	312	337	323	1001	689
% Set @ 100% el.		6.0%	3.8%	5.9%	4.5%	3.9%	4.3%	3.7%	3.8%	9.9%	4.7%
Caliper (mils)		74		59.3		46		103		94	
Delamination: Peel Strength (g)		316		>5000*		473		>5000*		>5000*	

\* Peel was greater than 5000 g; adhesive failure of tape in peel test occurred before cohesive failure of the composite nonwoven fabric.

#### EXAMPLES 6 AND 7

The fabrics of Examples 6 and 7 were made much like the Examples 4 and 5, with the inclusion of a layer of spunbonded nylon to act as a "stop" in the 5 fabric.

The composites were made by using the fiber blend of Examples 1-5, i.e.,

• 70 wt. % 2.2 dpf x 1.5" Type-182 Herculon® polypropylene staple fiber from Hercules,

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• 30 wt. % 2.0 dpf x 1.5" K-54  
copolyester/polyester bicomponent fiber from Hoechst  
Celanese Corp.

A four-layer laminate was formed consisting  
5 of:

- (1) a 33 g/m<sup>2</sup> carded layer of the above  
fiber blend,
- (2) a 170 g/m<sup>2</sup> rectangular, 18 x 9  
10 (strand/inch, MD x CD) elastic net  
stretched 100% in the MD,
- (3) a 10 g/m<sup>2</sup> layer of PBN-II<sup>®</sup> spunbonded  
nylon, and finally,
- (4) a 33 g/m<sup>2</sup> carded layer of the previous  
fiber blend.

15 The materials were entangled with the net in  
the stretched position (100% elongation in the MD).  
Both fabrics were then relaxed and dried. The fabric  
of Example 6 underwent no thermal treatment, and that  
of Example 7 was bonded in a double-wire through-air  
20 oven at 155°C.

The resulting material was gathered in the MD  
and had limited elasticity in the CD. It exhibited  
high extensibility in the MD, and good opacity as it  
extends. Once 100% elongation is reached, the PBN-II<sup>®</sup>  
25 spunbonded nylon acts as a "stop" to inhibit further  
elongation.

#### EXAMPLES 8 AND 9

Examples 8 and 9 were also made much like the  
Examples 4 and 5, but this time a polypropylene  
30 meltblown material was included in the structures.

The composites were made by using the fiber  
blend of Examples 1-7, i.e.,

- 70 wt. % 2.2 dpf x 1.5" Type-182 Herculon<sup>®</sup>  
polypropylene staple fiber from Hercules,
- 35 • 30 wt. % 2.0 dpf x 1.5" K-54  
copolyester/polyester bicomponent fiber from Hoechst  
Celanese Corp.



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A four-layer laminate was formed consisting of:

- (1) a 33 g/m<sup>2</sup> carded layer of the above fiber blend,
- 5 (2) a 170 g/m<sup>2</sup> rectangular, 18 x 9 (strand/inch, MD x CD) elastic net stretched 100% in the MD,
- (3) a 13 g/m<sup>2</sup> layer of meltblown polypropylene, and finally,
- 10 (4) a 33 g/m<sup>2</sup> carded layer of the previous fiber blend.

The materials were entangled with the net in the stretched position. In both examples, the fabrics were then relaxed and dried. In Example 8 the fabric  
15 was subjected to no thermal treatment, and Example 9 the fabric was bonded in a double-wire through-air oven at 155°C.

Properties of the fabrics of Examples 6 through 9 are set forth below. Examples 6 through 9  
20 again demonstrate how bonding gives a coherent composite. The un-bonded versions were easier to delaminate than the bonded version. Examples 6 and 8, unbonded composites, had peel strengths of 771 grams and 180 grams, respectively. Examples 7 and 9,  
25 through-air bonded composites, had peel strengths over 5000 grams (these samples also had an adhesive failure of the peel tape prior to any cohesive failure of the fabric). In addition, the bonded versions also demonstrated much higher tensile strengths than the un-  
30 bonded versions, but still had good stretch and recovery properties. Finally, as with previous examples, the bonded versions had much less fuzz and pilling of fibers.

Table 2 describes Examples 6-9 and also  
35 includes basic mechanical properties.

Table 2. Description and Properties of Examples 6 Through 9.

ITEM	EXAMPLE							
	6		7		8		9	
Pre-stretch	100% MD		100% MD		100% MD		100% MD	
Heat Treatment	none		through-air		none		through-air	
Additional Layer	PBN-II SB nylon		PBN-II SB nylon		MB polypropylene		MB polypropylene	
Strip Tensile Test:	MD	CD	MD	CD	MD	CD	MD	CD
Load (g/in) @ 25% el.	122	808	271	1025	109	199	188	324
Load (g/in) @ 50% el.	190	1578	663	2076	167	312	303	618
Load (g/in) @ 75% el.	272	1637	1475	2323	227	413	559	864
Load (g/in) @ 100% el.	579	1759	2640	2227	316	503	979	1055
Peak Load (g/in)	4120	1867	4873	2460	2211	2382	2762	1945
Elongation (%) at Peak	240%	91%	172%	85%	967%	968%	237%	254%
Cycle Test: Load (g/in) at 50 Elongation	Pull 1	197	1805	312	2429	198	319	421
	Pull 2	185	1387	255	1955	185	278	344
	Pull 3	181	1303	244	1820	185	264	319
% Set @ 50% el.	3.6%	3.7%	3.8%	4.2%	3.3%	3.8%	3.8%	4.1%
Cycle Test: Load (g/in) at 100% Elongation	Pull 1	705	1861	930	2655	391	507	1136
	Pull 2	575	1239	693	1540	341	399	888
	Pull 3	529	1040	655	1406	322	329	795
% Set @ 100% el.	3.5%	5.7%	5.4%	4.8%	3.7%	3.9%	4.4%	4.1%
Caliper (mils)	120		109		128		94	
Delamination: Peel Strength	771		>5000*		180		>5000*	

\* Peel was greater than 5000 g: adhesive failure of tape in peel test occurred before cohesive failure of the composite nonwoven fabric.

#### EXAMPLES 10-12: SCANNING ELECTRON MICROSCOPY

##### EXAMPLE 10

A rectangular, 25 x 15 (strands/inch, MD x CD) scrim was hydroentangled with a carded fiber blend  
5 of:

- 70 wt. % 1.5 dpf x 1.5" Type-54W Dacron® polyester staple fiber from Dupont,
- 30 wt. % 1.5 dpf x 1.5" polypropylene/polyethylene sheath/core bicomponent fiber from Chisso.

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The composite consisted of a sandwich with: a 31 g/m<sup>2</sup> layer of carded, blended fiber, 180 g/m<sup>2</sup> 25 x 15 elastic net, and an additional 31 g/m<sup>2</sup> layer of carded, blended fiber. The total fiber basis weight in the composite was 62 g/m<sup>2</sup>.

The web was entangled in the un-stretched state on a 31 x 25 (strand/inch) screen at 240 ft/min with two passes at 400 psi and four passes at 1400 psi for each side. The fabric was dried and through-air bonded in a double-wire through-air bonder at 155°C to form the final composite fabric.

Scanning electron microscopy indicated that the polyethylene sheath of the bicomponent fiber bonded to itself and also to the scrim. The polyester staple fiber was mobile and is not bonded to any elements of the composite.

#### EXAMPLE 11

A rectangular, 7 x 9 (strands/inch, MD x CD) scrim was hydroentangled with a carded fiber blend of:

- 70 wt. % 2.2 dpf x 1.5" Type-182 Herculon<sup>®</sup> polypropylene staple fiber from Hercules,
- 30 wt. % 3.0 dpf x 1.5" polypropylene/polyethylene bicomponent fiber from BASF.

The composite consisted of a sandwich with: a 31 g/m<sup>2</sup> layer of carded, blended fiber, 180 g/m<sup>2</sup> 7 x 9 elastic net, and an additional 31 g/m<sup>2</sup> layer of carded, blended fiber. The total fiber basis weight in the composite was 62 g/m<sup>2</sup>.

The web was entangled in the un-stretched state on a 31 x 25 (strand/inch) screen at 240 ft/min with two passes at 400 psi and four passes at 1400 psi for each side. The material was dried and through-air bonded in a double-wire through-air bonder at 155°C to form the final composite fabric.

Scanning electron microscopy indicated that the polyethylene sheath of the bicomponent fiber bonded to itself and also to the scrim. There was also some

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very light bonding between the polyethylene sheath and the polypropylene staple fiber, but the adhesion appeared to be poor and the bond-strength weak.

EXAMPLE 12

5 A rectangular, 18 x 9 (strands/inch, MD x CD) scrim was hydroentangled with a carded fiber blend of:

• 70 wt. % 2.2 dpf x 1.5" Type-182 Herculon® polypropylene staple fiber from Hercules,

10 • 30 wt. % 2.0 dpf x 1.5" K-54 copolyester/polyester bicomponent fiber from Hoechst Celanese Corp.

The composite consisted of a sandwich with: a 33 g/m<sup>2</sup> layer of carded, blended fiber, 180 g/m<sup>2</sup> 7 x 9 elastic net, and an additional 33 g/m<sup>2</sup> layer of carded, blended fiber. The total fiber basis weight in the  
15 composite was 66 g/m<sup>2</sup>.

The web was entangled in the un-stretched state on a 20 x 13 (strand/inch) screen at 60 ft/min with two passes at 400 psi and four passes at 1000 psi on one side, and then entangled on the other side with  
20 a 31 x 25 screen at the same entanglement conditions. The material was dried and calendered on a smooth-smooth calender at 100 ft/min, calender gap of 0.023", and calender roll temperature of 135°C for each roll.

Scanning electron microscopy indicated that  
25 the copolyester portion of the bicomponent fiber bonded to itself and not to the scrim. The polypropylene staple fiber was mobile and is not bonded to any elements of the composite.

A summary of these bonding results is shown  
30 in Table 3.

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Table 3. Summary of Bonding Results.

ITEM		EXAMPLE		
		10	11	12
Staple Fiber		polyester	polypropylene	polypropylene
Bicomponent Binder Fiber		polyethylene sheath polypropylene core	polyethylene sheath polyester core	copolyester sheath polyester core
Bonding of Binder Fiber to:	itself	yes	yes	yes
	staple	no	yes, but weak	no
	net	yes	yes	no

5

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## CLAIMS:

1. A composite elastic nonwoven fabric comprising:  
an elastomeric net having a plurality of apertures;  
5 a fibrous web comprising binder fibers and being intimately hydroentangled with said net such that at least a portion of said binder fibers extend through at least a portion of the apertures in said net; and  
at least a portion of said binder fibers  
10 having been thermally activated thereby bonding said hydroentangled web into a coherent, substantially unitary structure encompassing said elastomeric net.
2. The nonwoven fabric of Claim 1 wherein said fibrous web comprises binder fibers in an amount  
15 of between about 5 and about 50 wt. %.
3. The nonwoven fabric of Claims 1 or 2 wherein said fibrous web comprises fibers selected from the group consisting of polyester, polyolefin, nylon, acrylic, modacrylic, rayon, cellulose acetate, wool,  
20 cotton and/or wood pulp.
4. The nonwoven fabric of any of Claims 1 to 3 wherein said elastomeric net is substantially rectangular, and elastic in only one of the longitudinal or transverse directions.
- 25 5. The nonwoven fabric of any of Claims 1 to 3 wherein said elastomeric net is substantially rectangular, and is elastic in both the longitudinal and transverse directions.

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6. The nonwoven fabric of any of Claims 1 to 3 wherein said elastomeric net is of a non-rectangular geometry, and is elastic in one direction and inelastic in another direction.

5 7. The non-woven fabric of any of Claims 1 to 3 wherein said elastomeric net is of a non-rectangular geometry, and is elastic in all directions.

8. The nonwoven fabric of any of Claims 1 to 7 wherein said elastomeric net is maintained in a stretched condition during hydroentangling of said fibrous web and said net.

9. The nonwoven fabric of any of Claims 1 to 8 wherein said hydroentangled fibrous web and elastomeric net has been thermally treated in the stretched state.

10. The nonwoven fabric of any of Claims 1 to 8 wherein said hydroentangled fibrous web and elastomeric net has been thermally treated in the relaxed state.

20 11. The nonwoven fabric of any of Claims 1 to 10 additionally comprising a web of continuous filaments, bonded or unbonded, hydroentangled with said fibrous web and said net.

25 12. The nonwoven fabric of any of Claims 1 to 11 additionally comprising a meltblown web intimately hydroentangled with said fibrous web and said net.

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13. The nonwoven fabric of any of Claims 1 to 12 wherein said binder fibers are thermally activated at a temperature in the range of between about 100°C and about 135°.

5           14. The nonwoven fabric of any of Claims 1 to 13 wherein said elastomeric net comprises a thermoplastic elastomer.

15. The nonwoven fabric of Claim 14 wherein said elastomer is crosslinked.

10           16. The nonwoven fabric of any of Claims 1 to 14 wherein said elastomeric net comprises an A-B-A block copolymer, wherein A represents a styrene block and B represents unsaturated or fully hydrogenated rubber block.

15           17. The nonwoven fabric of any of Claims 1 to 16 wherein said elastomeric net additionally comprises a blended thermoplastic polymer.

18. The nonwoven fabric of any of Claims 1 to 17 wherein said binder fibers comprise polyethylene.

20           19. The nonwoven fabric of any of Claims 1 to 18 wherein said binder fibers comprise a polyester or copolyester polymer.

25           20. The nonwoven fabric according to any of Claims 1 to 19 wherein said binder fibers are bicomponent fibers.



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21. The nonwoven fabric according to any of Claims 1 to 20 wherein said binder fibers are present in said fibrous web in an amount of between 10 and about 40 wt. %.

5           22. The nonwoven fabric of any of Claims 1 to 21 wherein at least a portion of said binder fibers are thermally bonded to said elastomeric net.

          23. The nonwoven fabric of any of Claims 1 to 21 wherein there is substantially no bonding between  
10 said binder fibers and said elastomeric net.

          24. The nonwoven fabric of any of Claims 1 to 23 wherein said elastomeric net comprises between about 2 and about 30 strands per inch in each direction.

15           25. The nonwoven fabric of any of Claims 1 to 24 wherein said elastomeric net is substantially free from thermal disruption.

          26. The nonwoven fabric of any of Claims 1 to 25 wherein said binder fibers are thermally  
20 activated by calendering.

          27. The nonwoven fabric of any of Claims 1 to 26 wherein said binder fibers are thermally activated by through-air bonding.

          28. The nonwoven fabric of any of Claims 1  
25 to 27 further comprising a second fibrous web comprising binder fibers and being disposed on the opposed side of said net from said fibrous web and being intimately hydroentangled with said net such that at least a portion of said binder fibers in each of  
30 said fibrous webs extend through apertures in said net;

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at least a portion of said binder fibers in each of said webs having been thermally activated thereby bonding said hydroentangled first and second webs into a coherent, substantially unitary structure  
5 encompassing said elastomeric net.

29. The nonwoven fabric of Claim 28 wherein said second fibrous web comprises binder fibers in an amount of from about 5 to about 50 wt. %.

30. The nonwoven fabric according to any of  
10 Claims 28 and 29 wherein said second fibrous web comprises fibers selected from the group consisting of polyester, polyolefin, nylon, acrylic, modacrylic, rayon, cellulose acetate, wool, cotton, and/or wood pulp.

15 31. The nonwoven fabric of any of Claims 28 to 30 wherein said binder fibers are present in said second fibrous web in an amount of between about 10 and about 40 wt. %.

32. A process for producing a composite  
20 elastomeric nonwoven fabric comprising the steps:  
forming a layered structure comprising an elastomeric net having a plurality of apertures and a fibrous web comprising binder fibers;  
intimately hydroentangling said elastomeric  
25 net and said fibrous web sufficiently that at least a portion of said binder fibers in said fibrous web extend through apertures in said elastomeric net; and  
thermally treating said hydroentangled  
fibrous web sufficiently to thermally activate at least  
30 a portion of said binder fibers thereby bonding said hydroentangled web into a coherent, substantially unitary structure encompassing said elastomeric net.

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33. The process according to Claim 32 additionally comprising the step of stretching said elastomeric net in at least one of the longitudinal or transverse directions prior to said hydroentangling  
5 step; and maintaining said web in said stretched condition during said hydroentangling step.

34. The process of Claims 32 or 33 wherein said layered structure formed in said forming step additionally comprises a web of continuous filaments,  
10 bonded or unbonded.

35. The process of any of Claims 32 to 34 wherein said layered structure formed in said forming step additionally comprises a meltblown web.

36. The process of any of Claims 32 to 35  
15 wherein said fibrous web comprises between about 5 and about 50 wt. % binder fibers.

37. The process of any of Claims 32 to 36 wherein said fibrous web additionally comprises fibers selected from the group consisting of synthetic fibers,  
20 natural fibers and wood pulp fibers.

38. The process of any of Claims 32 to 37 wherein said thermal treating step is conducted by passing said hydroentangled web through heated calender rolls.

25 39. The process of Claim 38 wherein said heated calender rolls are smooth calender rolls.

40. The process of any of Claims 32 to 37 wherein said thermal heating step is conducted by passing said hydroentangled web through a through-air  
30 oven.

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41. The process of any of Claims 32 to 40 wherein said thermal heating step is performed with the web in a stretched state.

42. The process of any of Claims 32 to 40 wherein said thermal heating step is performed with the web in a relaxed state.

43. The process of any of Claims 32 to 42 wherein said binder fibers are present in said fibrous web in an amount of between about 10 and about 40 wt. %.

44. The process of any of Claims 32 to 43 wherein said elastomeric net comprises about 5 to about 30 strands per inch in each direction.

45. The process of any of Claims 32 to 44 wherein said layered structure formed in said step of forming a layered structure comprises said elastomeric net sandwiched between said fibrous web and a second fibrous web.

46. The process of Claim 45 wherein binder fibers are present in said second fibrous web in an amount of between about 5 and about 50 wt. %.

47. The process of Claims 45 or 46 wherein said second fibrous webs includes fibers selected from the group consisting of synthetic fibers, natural fibers and wood pulp fibers.

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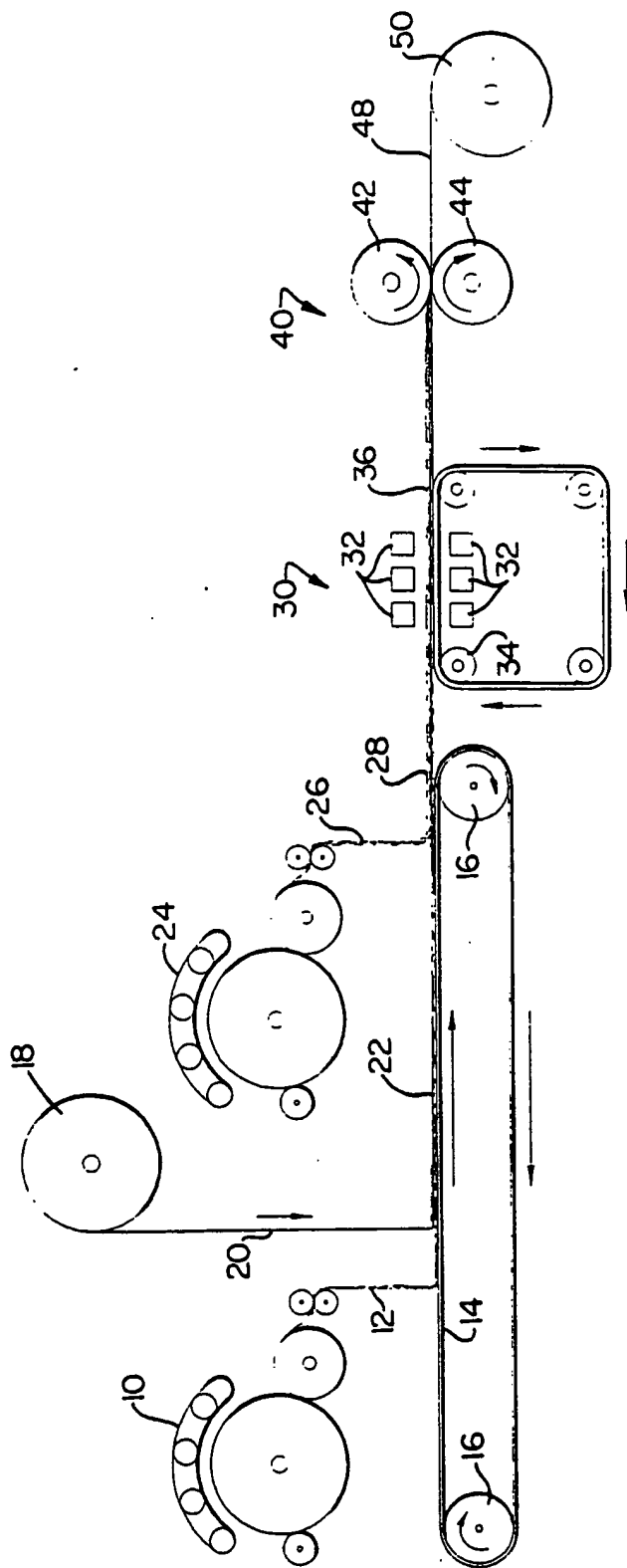


FIG. 1.

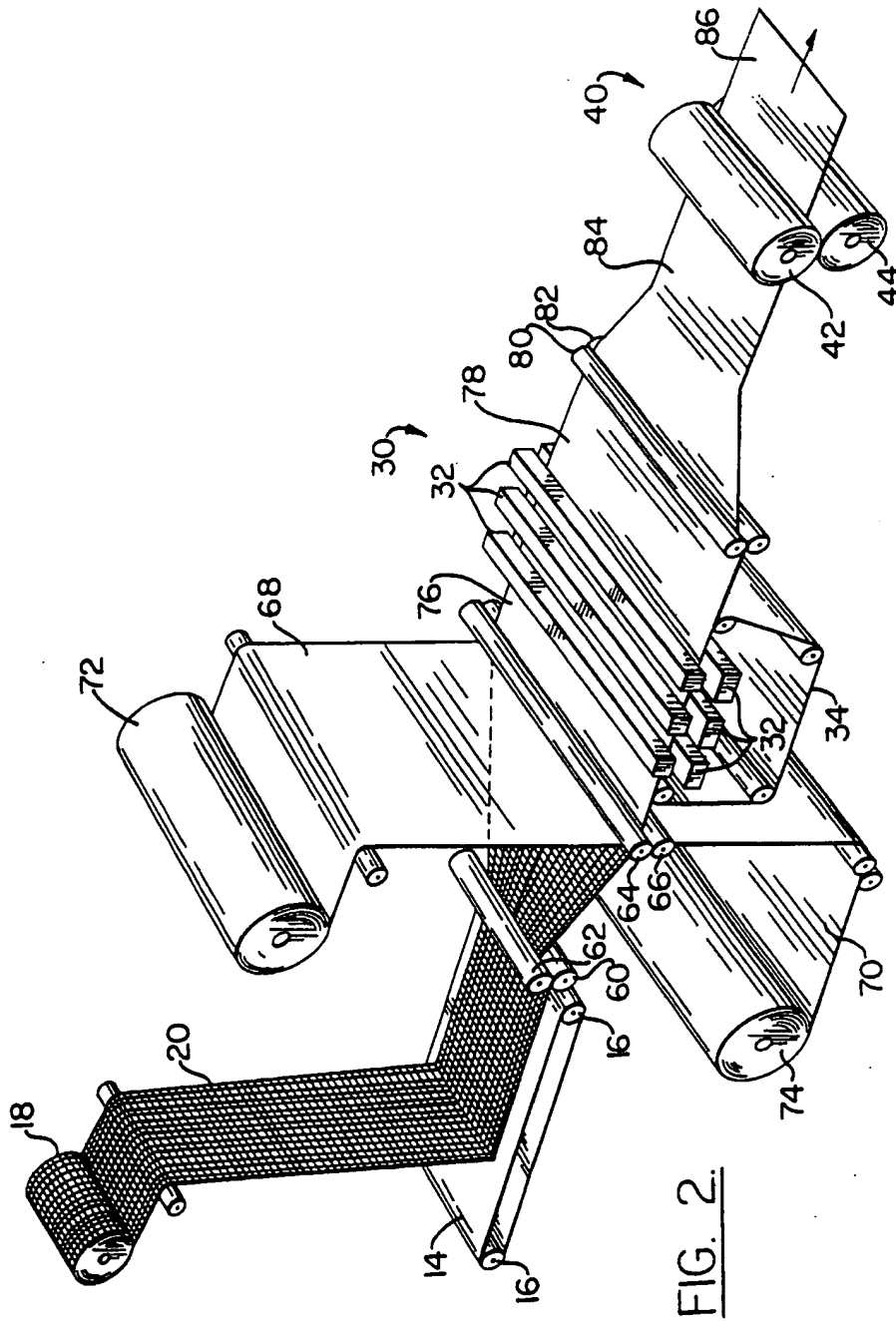


FIG. 2.

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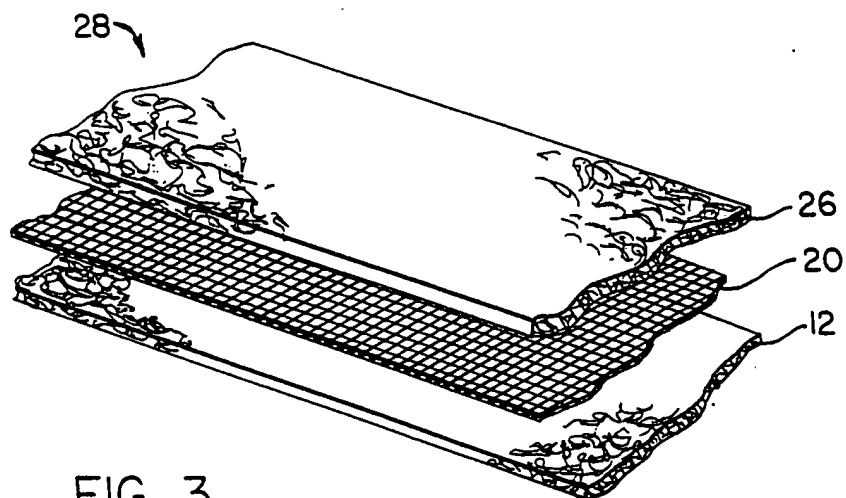


FIG. 3.

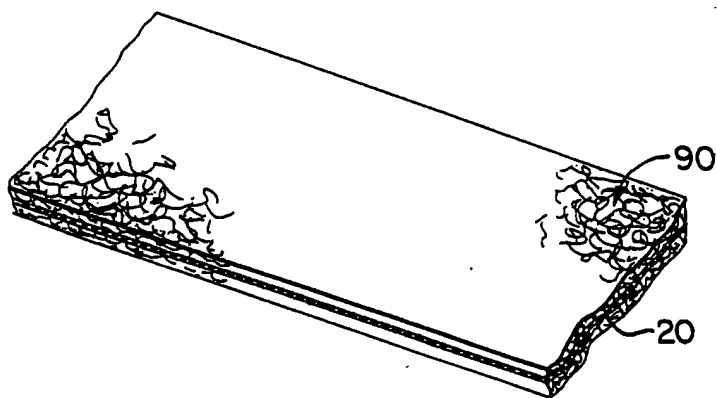


FIG. 4.

SUBSTITUTE SHEET

## INTERNATIONAL SEARCH REPORT

PCT/US 93/00567

International Application No

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (If several classification symbols apply, indicate all) <sup>6</sup>		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. 5 D04H1/48; D04H13/00; D04H1/70		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched <sup>7</sup>		
Classification System	Classification Symbols	
Int.Cl. 5	D04H	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched <sup>8</sup>		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT<sup>9</sup></b>		
Category <sup>10</sup>	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
A	EP,A,0 007 802 (TEIJIN LTD) 6 February 1980	1,3,4,6, 13-16, 32,37,38
A	see whole document	
A	DE,A,2 364 958 (IMPERIAL CHEMICAL INDUSTRIES LTD.) 17 July 1975	1,2,3, 11,13, 20-22, 27-32, 36,37,40
A	see example 1	
A	US,A,4 863 779 (DAPONTE) 5 September 1989	1,4,7, 9-12,14, 16,17, 32-35, 37,38, 45,47
	see whole document	
<p><sup>10</sup> Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"A" document member of the same patent family</p>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search		Date of Mailing of this International Search Report
16 APRIL 1993		02 05 93
International Searching Authority		Signature of Authorized Officer
EUR PEAN PATENT FFICE		VAN BEURDEN-HOPKINS



III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	US,A,4 775 579 (HAGY ET AL.) 4 October 1988 cited in the application see whole document -----	1,3,5,7, 8,11,14

**ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO.**

US 9300567  
SA 69793

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on  
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16/04/93

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A-0007802	06-02-80	JP-A- 55098956	28-07-80
		JP-C- 1370785	25-03-87
		JP-A- 55021420	15-02-80
		JP-B- 61037380	23-08-86
		US-A- 4296163	20-10-81
DE-A-2364958	17-07-75	None	
US-A-4863779	05-09-89	AU-A- 7049587	01-10-87
US-A-4775579	04-10-88	EP-A- 0315507	10-05-89
		JP-A- 1148856	12-06-89

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82